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New Data on the Structure of the Nucleus

Photonuclear Conference Surveys Recent Work on Radiation-Induced Reactions

A COMPREHENSIVE review of recent advances in knowledge of the structure and internal forces of the atomic nucleus, as revealed by studies of nuclear reactions induced by X- and gamma rays, was presented at the April 30 to May 1 sessions of the Sixth Photonuclear Conference. Meeting at the National Bureau of Standards, the conference was attended by scientists from leading centers of photonuclear research in North and South America, Europe, and Australia. Sponsors of the meeting were the U. S. Atomic Energy Commission, the Office of Naval Research, the Air Force Office of Scientific Research, and the Bureau.

Photonuclear research is concerned with essentially the same problems of nuclear structure as other areas of low-energy (i. e., below 100 Mev or so) nuclear physics. However, the photonuclear scientist must face a special set of experimental difficulties. Because sources of monoenergetic photons are difficult to obtain, it has usually been necessary to work with continuous (bremsstrahlung) spectra whose shape and absolute scale are not too accurately known. As a result, in most experiments the cross sections must be obtained by analysis of measurements made at many different peak-bremsstrahlung energies. For this reason, much interest centers on the monitoring of bremsstrahlung beams and calibration of the accelerators—betatrons and synchrotrons, for example—that produce the photons by bombarding a high-Z (i. e., high-atomic number) target with an electron beam.

It was to discuss such problems and to compare experimental methods and results that the first Photonuclear Conference met—somewhat informally—several years ago; and its success in promoting the solution of experimental problems has led to its repetition every year or two since.

An extensive survey of current experimental activity was given on the first day of the present conference, when 21 laboratories reported on their general programs and recent experimental results. The second day of the conference was held in conjunction with the Washington Meeting of the American Physical Society. The morning session was devoted to invited papers by G. E. Brown (Institute of Theoretical Physics, Copenhagen), J. H. D. Jensen (Univ. of Heidelberg), R. E. Marshak (Univ. of Rochester), and J. H. Smith (Univ. of Illinois). In the afternoon, contributed papers to the Physical Society on photonuclear reactions and bremsstrahlung spectra were read.

The conference discussions may be conveniently grouped under four headings: Fundamental reactions, reactions in light nuclei, reactions in heavy nuclei, and experimental techniques.

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Fundamental Reactions

Photonuclear reactions (exclusive of meson production) that might be classified as "fundamental" are the elastic scattering of photons by hydrogen and deuterium and the photodisintegration of deuterium and helium. Few new measurements on these reactions were presented. Data on the photodisintegration of helium are gradually becoming available and improvements in experimental technique indicate that the evident need for extensive measurements of this kind may be met in the near future.

Advances in the theory of the photodisintegration of deuterons were presented in R. E. Marshak's invited paper. Previous calculations have usually neglected some of the terms for the dipole contributions (e. g., outgoing F waves and effects of noncentral forces); while reasonably successful up to about 20 Mev, they failed markedly at higher energies. By including all these terms, using the Signel-Marshak potential that has been so successful for (p, p) [1] scattering up to about 150 Mev, the University of Rochester group obtained good agreement between theory and experiment for the deuterium reaction up to about 75 Mev. At higher energies, a need for additional terms, probably including specific mesonic effects, seems indicated.

The apparent success of this theory, together with improved absolute-monitoring techniques, should act as a powerful stimulus to the experimentalists. Further encouragement, especially for the interesting and difficult polarization experiments, was provided by the reports on measurements of polarization in bremsstrahlung beams presented by R. W. Kenny (Univ. of California) and P. Axel (Univ. of Illinois).

Reactions in Light Nuclei

One of the more interesting developments concerning the light nuclei has been the accumulation of evidence for the existence of sharp breaks in bremsstrahlung activation curves. The clear implication is that the "breaks" correspond to well-defined nuclear-energy levels. From the shapes of the curves (neutron yield versus peak-photon energy), estimates have been made of the level-width and peak cross sections.

Several attempts to confirm the existence of such levels were reported. These include measurements of total absorption coefficients (Univ. of Pennsylvania), high-resolution measurements of the energy spectrum of protons from (γ, p) reactions (Stanford Univ.), and high-resolution measurements of the cross section for the inverse (p, γ) reactions (Australian National Univ.). Little indication for level structure was found; though it appears likely, in most of these exeriments, that the resolution was just marginal for observing structure in the energy regions studied.

The Stanford University and NBS reports, given by W. C. Barber and H. W. Koch, respectively, showed that measurements of (γ, p) reactions by counter techniques have improved considerably in ease and accuracy. The strong influence of quadrupole and higher modes of photon absorption at higher energies is quite evident from the asymmetry in the angular distributions. Data of this kind are available from many laboratories, particularly for carbon-12; the data are in fair agreement, except perhaps for absolute scale, and such disagreements will probably be reduced as analysis of the experimental results is refined. The abundant information on these reactions provides shell-model theoreticians with an opportunity for rather detailed tests of shell-model wave functions.

The high-energy photoeffect was well summarized in J. H. Smith's paper on the "pseudo-deuteron" model. According to this theory, at energies above about 100

Scientists attending Photonuclear Conference enjoy a box lunch in the betatron laboratory during an informal session on giant-resonance width. (Photo courtesy of *Life*.)

J. H. D. Jensen (Univ. of Heidelberg, standing left) and G. E. Brown (Institute of Theoretical Physics, Copenhagen, standing right) discuss a point of photonuclear theory at the Conference. (Photo courtesy of *Life*.)

Mev, the primary source of high momentum for emitted particles is in the collisions of protons and neutrons inside the nucleus. When the incident photon (which carries very little momentum itself) interacts with a proton and neutron that are undergoing collision within the nucleus, the result is analogous to the interaction of a photon with a deuteron. There should thus be correlated emission of neutrons and protons, an effect that has been observed and studied extensively at MIT and Illinois. The work at Illinois, described by Smith, indicates that, if scattering of neutrons and protons on leaving the nucleus is taken into account, above 100 Mev, essentially all of the photoeffect can be explained in terms of the cross section for photodisintegration of the deuteron. By examining the angular correlation between the neutrons and protons, a measure is obtained of the momentum distribution within the nucleus.

Reactions in Heavy Nuclei

Reports on reactions in heavier elements were almost completely dominated by studies of the influence of nuclear deformation—deviation from spherical symmetry—on the giant resonance.[2] The prediction that the giant resonance should be split had been made independently by Okomoto and by Danos. At the conference, D. H. Wilkinson (Oxford Univ.) presented calculations based on a single-particle model which also predict splitting of the giant resonance. Perhaps easiest to visualize is the theory of Danos, based on the hydrodynamic nuclear model of Goldhaber and Teller. In this model the frequency of nuclear oscillation (which corresponds to the energy of the giant-resonance peak) depends on the nuclear dimensions. For a spherical nucleus, one resonance should be observed, corresponding to the nuclear diameter. For a deformed nucleus, there should be two resonances, corresponding to the major and minor axes; in a highly deformed nucleus like Ta, the two resonances should be clearly distinguishable.

Attempts to test these predictions were made by laboratories at the universities of Melbourne, Pennsylvania, and Saskatchewan, the Australian National University, and the National Bureau of Standards. Most of the results could not resolve the two peaks, but the correlation between over-all width of the giant resonance and nuclear deformation appeared quite definite. Data presented by H. W. Koch, obtained with extremely good statistical accuracy, showed a pronounced splitting for Ta and virtually none for Au. Nuclear quadrupole moments (which measure deviation from sphericity) deduced from these data are in excellent agreement with values obtained by Coulomb-excitation experiments. Study of giant-resonance splitting would seem a valuable means of determining quadrupole moments; for spin-0 nuclei it is perhaps the only way.

Theories of the giant resonance that apply primarily to heavier nuclei were presented in the invited papers of G. Brown and J. H. D. Jensen. Jensen discussed the semiclassical description of the giant resonance, emphasizing the general results that are given by this theory and must be contained in any proper theory. Brown dealt mainly with the anomalously large photoproduction of fast particles from heavier nuclei. An explanation had been given by Wilkinson, on the basis of a modified shell model. Brown has attempted to refine this formulation by using nuclear dispersion theory. The results are in general agreement with experiment and with the Wilkinson calculations.

Experimental Techniques

Studies of the reliability of betatron energy-control systems were reported by J. Halpern (Univ. of Pennsylvania), L. Katz (Univ. of Saskatchewan), J. McElhinney (Naval Research Laboratory), and A. S. Penfold (Univ. of Chicago). Tests described by Halpern indicate that, with the usual means of energy control, the betatron at the University of Pennsylvania becomes inaccurate above 20 Mev. This is believed due to saturation effects in the magnet-core iron; and a new energy-control system has been constructed which samples the magnetic field at the X-ray target. If the electrons are not expanded into the target too rapidly, this system is expected to permit improved measurement of the betatron energy.

Efforts to develop an electron-spin resonance device for absolute calibrations of accelerator energy at the National Bureau of Standards were reported; this device should be particularly useful for circular accelerators. A new radiation monitor for the Bureau's Betatron Laboratory was also discussed. Calibrated over a fairly large energy region with an accuracy of a few percent, the monitor will increase reliability of cross-section measurements—especially on the high-energy side of the giant resonance.

Exciting possibilities were suggested by the account given of a 32.5-Mev, 4-kw linear accelerator soon to be installed at General Atomics Laboratory. Described by C. Oxley (General Dynamics Corporation), the new machine is designed to produce X-ray and electron-beam intensities several orders of magnitude higher than those of existing accelerators.

Photonuclear research has, for several years, consisted largely of what might be called "survey experiments"—where the main concern is with the systematics of nuclear reactions and measurements are made of total cross sections, location of the giant-resonance peak, etc. The conference discussions show that this survey period is nearly over and that the emphasis is shifting toward more accurate investigation of the giant-resonance shape and of detailed features of the partial cross sections to various excited states.

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[1] *The symbol "(x, y)" represents a reaction in which a nucleus is bombarded by an x particle and emits a y particle; the letters p, n, and γ, which (among others) may be substituted for the x and y, stand for proton, neutron, and gamma-ray photon, respectively.*

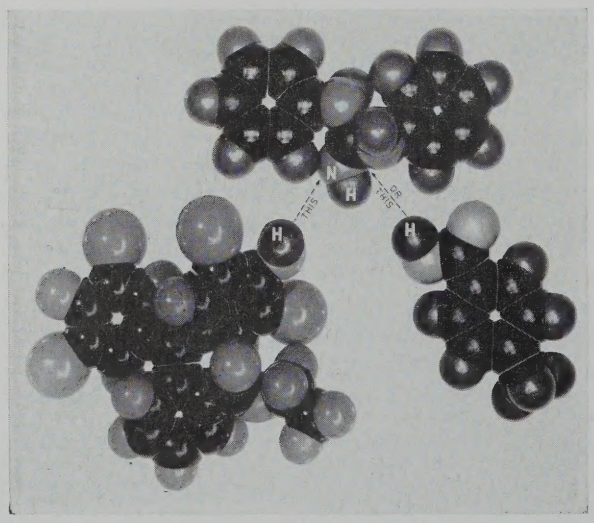
[2] *A general feature of photonuclear reactions is that the cross section (probability that the reaction takes place) is greatest for relatively low energies. Thus the cross sections of (γ, n) and (γ, p) reactions rise to a peak near 20 Mev and then fall off rapidly. This peak is the "giant-resonance".*

ACID STRENGTHS IN BENZENE

THE RELATIVE STRENGTHS of 40 carboxylic acids in benzene [1] have been measured and correlated with molecular structure by the Bureau in a study partly supported by the Air Force Office of Scientific Research. This work was part of a systematic investigation of acidity and basicity in nonaqueous solutions directed by Marion Maclean Davis of the physical chemistry laboratory. Results of the present study show that the acid reaction is governed not only by the nature of the substituted groups and their proximity to the carboxyl group (–COOH), but also by the nature of the solvent. The data have been compared with previously published values for relative strengths in water, as well as in several nonaqueous solvents.

In recent years, accelerated experimentation and development in the chemical industry have brought to the forefront many organic solvents previously unknown or not in general use. Acetic acid, bases like pyridine and dimethylformamide, halogenated hydrocarbons, alcohols, ethers, ester, and still newer types of solvents are now in use. A need has consequently arisen for a better knowledge of the influence that different types of solvents have on interactions of dissolved materials, such as acids and bases. The comparative inertness of benzene makes it one of the best solvents to use in studying the nature of interactions between dissolved acids and bases, including various manifestations of hydrogen bonding. These interactions are masked in water because the water itself is associated in various ways with the solutes. Knowledge of acid-base behavior in benzene helps in interpreting the effects of different media.

A spectrophotometric technique was used to measure the strength of the dissolved acids. Before this technique was developed, no procedure existed for determining the relative reactivities of protonic acids, such as benzoic-acid derivatives, when dissolved in benzene and similar solvents. The only criteria for relative strengths of the protonic acids were the constants for ionic dissociation, which were measured in water or similar solvents such as alcohols. Generally, the relative strengths of these acids were assumed to be independent of the solvent. However, it has since been found that the relative strengths of acids in organic solvents may differ from their relative strengths in water. The Bureau began systematic studies of acid-base reactions in nonaqueous solvents in 1941. Initiated at the request of military and industrial representatives, the program has included the investigation of many types of indicators that show the presence of acids and bases in benzene and other hydrocarbons by vivid color changes, selection of the most suitable indicators for specific acids and bases, and the evaluation of methods for measuring total and relative acidity and basicity.



Competitive association of the indicator, bromophthalein magenta *E* (lower left), and an aromatic acid, *p*-toluic acid (lower right), with the same base (1,3-diphenylguanidine) (top). The relative strengths of the aromatic acid and the indicator are measured by their relative tendencies to release protons to the nitrogen of the base.

Spectrophotometric Method

In the spectrophotometric procedure for measuring relative strengths of carboxylic acids in benzene, two commercially available reagents are used: bromophthalein magenta *E* (3′,3″,5′,5″-tetrabromophenolphthalein ethyl ester) and 1,3-diphenylguanidine. Mixtures containing known concentrations of these two reagents and the carboxylic acid under study are prepared and the spectral absorption of each mixture is measured. From color changes which occur in the visible region of the spectrum, the association constants ($K_{assoc.}$) indicating the tendencies of the various carboxylic acids to associate with diphenylguanidine, are calculated. The relative strengths of the organic acids are expressed by these equilibrium constants or their logarithms.

Carboxylic acids in benzene give a colorless solution in the visible spectral region where the data are collected, as does 1,3-diphenylguanidine and the various salts formed by association of the acids with diphenylguanidine. In contrast, bromophthalein magenta *E* gives a yellow solution in benzene, which changes to magenta when the indicator combines with diphenylguanidine; the color then reverts toward yellow upon addition of one of the benzoic acids, because of the acid's competitive association with diphenylguanidine.

Results

Measurements using the spectrophotometric method were made on benzoic acid, 31 of its monosubstituted derivatives (including all the benzoic acids substituted in the ortho, meta, or para position with $-CH_3$, –OH, $-OCH_3$, $-NH_2$, $-NO_2$, or the halogens), and eight di- or trisubstituted benzoic acids.

The results show that a change from water to benzene does not affect the order of the strengths of most meta-substituted carboxylic acids. That is, the strengths of the meta-substituted acids remain the same in relation to each other although occasional minor variations occur. Thus, when a graph is constructed by plotting the strengths of acids in water, expressed as pK ($-\log K_{dissoc.}$) values, against the corresponding values of $\log K_{assoc.}$ in benzene, almost all the points for the meta-substituted acids fall on a straight line, as does that for benzoic acid itself. This straight line can be adopted as a "norm" of acidic behavior in the two solvents. It then follows that an experimental point located above the line indicates an acid of abnormal strength in water or abnormal weakness in benzene, or both; for points located below the line the reverse is true.

Para-substituted benzoic acids are found to lie fairly close to the norm. However, there are a few exceptions: For example, the point for *p*-hydroxybenzoic acid occurs a little below the line, while the point for *p*-nitrobenzoic acid occurs above the line. The deviations seem most likely to be caused by association of water molecules with the substituent group.

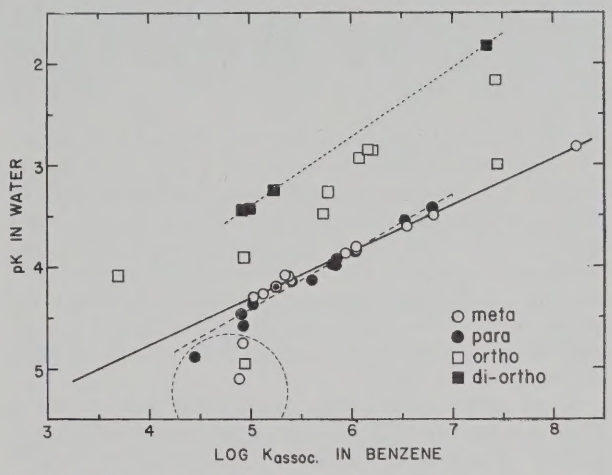
One of the most significant findings of the study is the pronounced effect on most ortho-substituted acids when the solvent is changed from water to benzene. Although *o*-toluic acid (*o*-methylbenzoic acid) exhibits anomalously high acidity in water, it shows normal acidity in benzene; that is, it is weaker than its meta and para isomers. This result agrees with the order of strengths predictable, considering the electron-attractive nature of the methyl group and its relative proximity to the carboxyl group in the three isomers.

The explanation most generally favored for the exceedingly high strength of ortho-substituted benzoic acids in water is steric inhibition of resonance of the carboxyl group with the benzene ring. An acid-weakening resonance of the carboxyl group with the aromatic ring tends to occur whenever these two groups can maintain a coplanar or nearly coplanar alinement. In the past, the high strength of *o*-toluic acid in water has been attributed solely to the bulky methyl group; however, the Bureau study has shown that bulkiness of the methyl group is not the only cause. Evidently, inhibition of resonance is also increased by attachment of water molecules to the carboxyl group through hydrogen bonds. Whatever may be the actual role of these water molecules in augmenting the strength of *o*-toluic acid, the same factor must be partly responsible for other cases of abnormally high strength of carboxyl acids when in aqueous solutions.

In some cases, reductions in the apparent acidic strength when the solvent is changed from water to benzene are attributed to the additional factor of intramolecular hydrogen bonding (chelation). An extreme example is *o*-methoxybenzoic acid, which is much weaker in benzene than in water because of the strong attraction of the carboxylic hydrogen to the methoxy oxygen. A similar but weaker tendency toward hydrogen bonding probably exists when a halogen atom is present in the position ortho to the carboxyl group.

Only four di-ortho-substituted benzoic acids, those with substituent groups $-CH_3$, $-OCH_3$, or $-Cl$, were measured in benzene. The strengths of all are linearly related in benzene and water although they are too weak in benzene, or too strong in water, (or both) to conform to the "norm".

The strengths of all four of the aminobenzoic acids studied (that is, anthranilic acid, its *m*- and *p*-isomers, and *m*-dimethylaminobenzoic acid) when referred to this norm were found to be greater in benzene than in water.



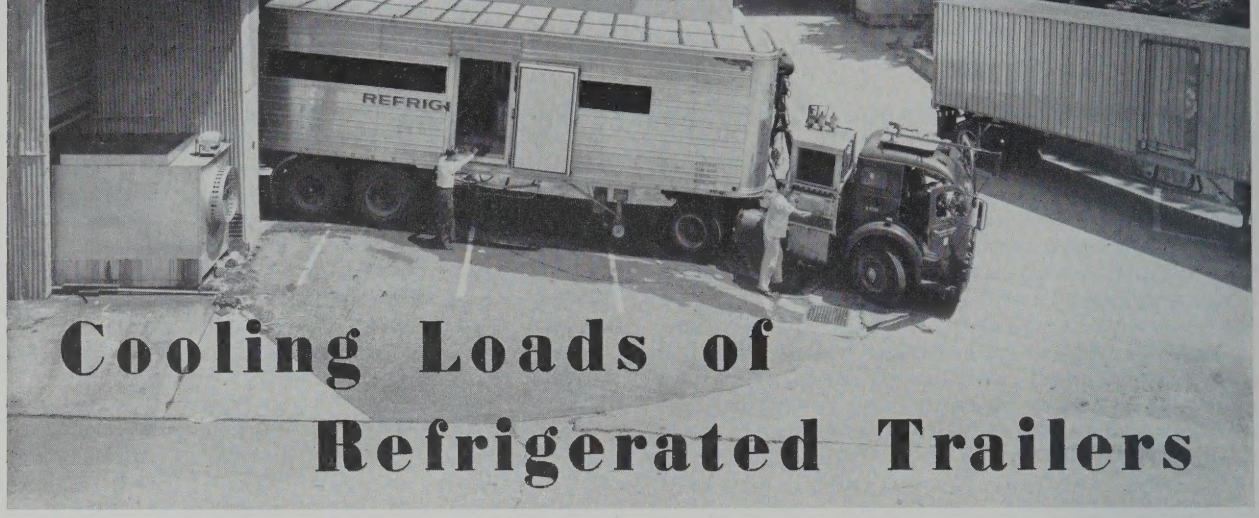
Relationship of the acidities of some substituted benzoic acids in water and in benzene, expressed respectively in terms of pK (that is, $-\log K_{dissoc.}$) and $\log K_{assoc.}$ The relative strengths of most meta- and para-substituted acids are not influenced by a change in solvent.

Applications of the Method

The method used to measure relative acidic strengths in benzene should be applicable to other colorless inert solvents if the reagents are soluble enough in the solvent concerned. When the acids are comparable in strength to benzoic acid, bromophthalein magenta *E* is a satisfactory acidic indicator. If competitive associations are to be minimized in measuring $K_{assoc.}$, the concentrations of the three reagents must be kept small as well as comparable in magnitude. When carboxylic-acid molecules are present in high concentrations, they tend to associate in pairs (dimers). In dilute solutions, mainly single molecules seem to be present. A somewhat more detailed study of benzoic acid has demonstrated the possibility of measuring the dimer-monomer equilibrium constants (K_{21}) for such carboxylic acids, as well as the $K_{assoc.}$ values.

The Bureau is extending its investigations in this general area to other types of acids and bases as well as to different temperatures.

[1] *For further technical details, see* The relative strengths of forty aromatic carboxylic acids in benzene at 25° C, *by M. M. Davis and H. B. Hetzer, J. Research NBS* **60**, *569 (1958) RP2871. A summary of earlier studies may be found in* Acidity and basicity in organic solvents, *NBS Tech. News Bul.* **36**, *168 (1952).*



Cooling Loads of Refrigerated Trailers

A METHOD for determining the cooling load of refrigerated trailers is currently under study at the Bureau in a program sponsored by the Department of of Agriculture, the Quartermaster Research and Engineering Command, and the Truck-Trailer Manufacturers Association. The objective of this cooperative project is the development of a standard laboratory rating method that will adequately simulate the heat-transfer conditions under which trailers are actually operated on the road.

Work on the project is now well under way. Cooling loads of typical trailers have been compared in the laboratory and on the road, methods are being devised to simulate the effects of solar radiation and wind pressure, and air- and moisture-transfer processes through trailer walls are being investigated. Results so far indicate that any laboratory method must include a means for simulating the air leakage that occurs on the road; significant reductions in cooling load would be realized if air leakage could be eliminated.

At present, there is no standard method in common use to accurately determine the cooling load of a refrigerated trailer. An accurate method would enable the trailer manufacturer to establish a rating for each of his models; and the purchaser, knowing this rating, the product load contributed by the commodity being carried, the length of haul, and certain environmental conditions, could then select the most economical and efficient refrigerating equipment for his needs. The method under development by P. R. Achenbach and C. W. Phillips of the refrigeration laboratory promises to provide the manufacturer with a reliable technique for determining these ratings.

Equipment and Procedure

Three 35-ft refrigerated semitrailers were chosen for the laboratory and road tests. The laboratory consisted of an insulated structure specially equipped with humidity and temperature-control facilities. The trailers were placed inside this building, on three calibrated platform scales to determine the weight gain caused by condensation of infiltrated moisture. An adjoining room was equipped with suitable controls and instruments. Outside the test structure was installed a prototype comparison heat-sink apparatus. This consisted of sectionalized, semiportable, refrigerating equipment which included three motor-driven two-speed compressors with water-cooled condensers, two brine chillers, and a metered-heat comparator.

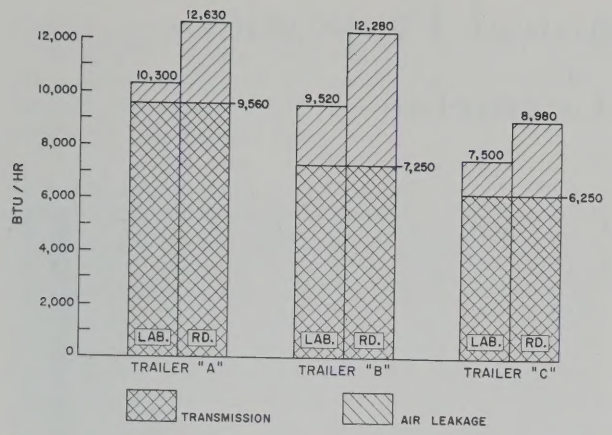
Test site at the Bureau, where method for determining the cooling loads of refrigerated trailers is being studied. Specially equipped tractor and trailers were used for the road tests.

A refrigeration coil with an integral blower was placed inside the trailer at approximately the same height above the floor as in typical trailer-refrigerating units. When needed, an electric-space heater was used to counteract the excess-cooling capacity of the refrigerating units.

Thermocouples were installed inside and outside the trailer near the walls to measure the average interior and ambient temperatures. Several large fans circulated the air around the trailer to promote uniform ambient temperature and humidity within the test structure. Controls and equipment were set and adjusted to maintain the selected standard-test condition, 0° F inside the trailer and 100° F and 50-percent relative humidity outside the trailer.

The laboratory tests of each trailer were made to determine the cooling load and simultaneous gain in weight due to the accumulation of water or ice under the standard test conditions. Chilled brine was circulated at a constant rate through the cooling coil inside the trailer and around an electric heater immersed in the brine circuit outside the trailer. The temperature rises produced in the brine by the heat absorbed inside the trailer and by the electric heater were measured as well as the power used by the electric heater. By equating the ratios of the two temperature rises and the two heat-energy sources, the unknown cooling load of the trailer could be determined.

For the road tests, carried out at 50-mph average road speed, a tractor was altered to carry a two-stage gasoline-engine-driven refrigerating system and an electric generator, also gasoline-engine driven. Controls and instrumentation for measuring the cooling load of the trailers were mounted inside the cab. The cooling load was determined by observing the rate of chilled-brine circulation through the cooling coil, the temperature rise of the brine inside the trailer, and the electric energy dissipated inside the trailer by fans and other equipment.



Comparative laboratory and road heat gains for three trailers, showing computed values of heat transfer caused by air leakage and transmission in each case.

Test Results

Comparison of laboratory and road tests shows that the heat gain of a refrigerated trailer was significantly greater during road operation than under stationary laboratory conditions for the same ambient temperature and humidity. This increase ranged from about 20 to 30 percent for the three trailers at ambient conditions of 100° F dry-bulb temperature and 50-percent relative humidity (R. H.). Since these ambient conditions were not attained during the road tests, this comparison is based on extrapolation of the observed data to the standard-laboratory test conditions. The extrapolated heat-gain values of the three trailers for these conditions ranged from 9,000 Btu/hr to 12,600 Btu/hr for a road speed of 50 mph.

The increase in heat gain on the road was due principally to air leakage into the trailer construction under the impact pressure of the air against the front of the trailer. Additional small increases were caused by solar radiation and the movement of air, heated by the engine, under the floor of the trailer.

The air leakage and ice accumulation in the trailers were not negligible even under stationary conditions in the laboratory. The weight-gain rates caused by ice accumulation averaged 0.32, 0.54, and 0.98 lb/hr for the three trailers during the laboratory tests. By assuming that the leakage air entered the trailer walls at ambient conditions of temperature and humidity and left the trailer body saturated at 0° F, the air leakage rate and the heat transferred to the cooling unit by the air leakage was computed for each trailer from the rate of weight gain. On this basis, the minimum air-leakage rates in the laboratory were 235, 395, and 715 ft^3/hr and the corresponding values of heat transfer caused by the air leakage were 740, 1,250, and 2,270 Btu/hr.

If the heat-transmission-rate per degree-temperature difference on the road, exclusive of heat transferred by air leakage, were assumed to be the same as in the laboratory, the air leakage could be deduced for the road tests. This procedure results in minimum air-leakage rates for the road tests of 970, 860, and 1,590 ft^3/hr and corresponding heat-transfer rates of 3,070, 2,730, and 5,030 Btu/hr caused by air leakage at the standard-test conditions. On this basis, the heat gain due to air leakage ranged from 32 to 69 percent of the transmission-heat gain. These percentages indicate that significant reductions in cooling load could be achieved by eliminating air leakage in trailer bodies.

The effects of solar radiation on a trailer are largely nullified by the rapid-air motion over the vehicle at 50 mph. In a typical test at this road speed in bright sunshine, incident solar radiation raised the surface temperature of the roof and one side of the trailer about 7.5° F above ambient air temperature. On a weighted-average basis this corresponds to an approximate 3-deg rise in temperature for the entire exterior surface. When the trailer was sitting still, the sun sometimes raised the roof temperature by as much as 25 deg.

The underside of the trailer warmed up as much as 15° above ambient temperature during road operation, principally by waste heat from the tractor engine. This rise would not cause a very large increase in over-all heat gain of the trailer, but could affect the preservation of food on the interior floor of the trailer unless chilled air were circulated around and under the load.

At 50 mph, impact air pressures up to 1.25 in. water gage (W. G.) occurred on the nose of a trailer where it was not shielded by the tractor, although the average pressure over the entire front end of the trailer was probably considerably below this value. The static pressures in the cargo space, in the insulation space, and over most of the exterior skin surface (excluding the nose) were about equal and ranged from 0.2- to 0.4-in. W. G. below the pressure of the undisturbed atmosphere as measured ahead of the vehicle. The leakage air probably entered the trailer body primarily on the nose of the trailer and left the body over the remainder of the surface. Most of the moisture in the ambient air was deposited as ice in the insulation space. The air exchange between the insulation space and the outdoors was probably several times greater than between the cargo space and outdoors.

The results suggest that smaller refrigerating units could be used if air leakage could be eliminated; alternatively, less insulation would be required if air leakage were significantly reduced. Moreover, if moisture could be kept out of the insulation spaces, deterioration of trailer bodies would proceed more slowly.

Tractor, showing the refrigerating equipment, some of the brine-circulating equipment on the front of the trailer, and humidity, temperature, and solar-radiation instruments on the roof of the tractor.

Determining Mechanical Properties of Brittle Cermets

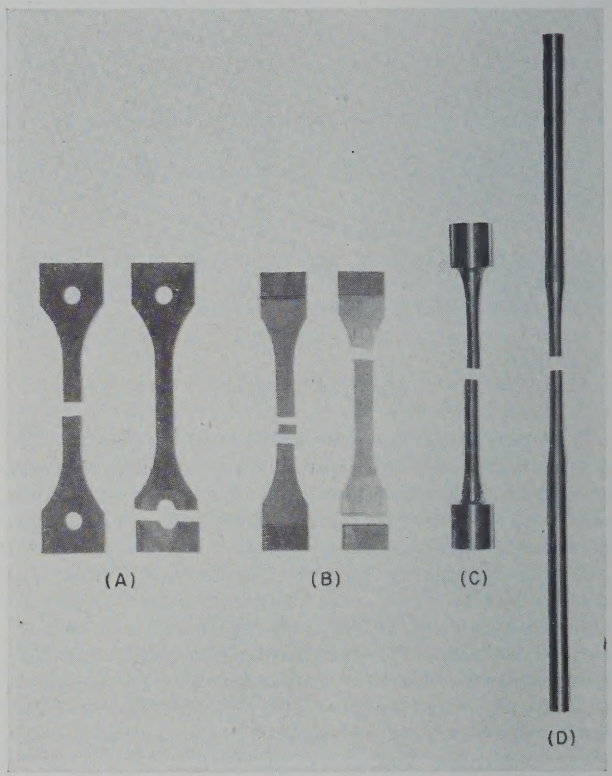
METHODS generally used to determine the mechanical properties of ceramics have been evaluated by the Bureau at the request of the Army Watertown Arsenal.[1] The evalaution was needed because present uncertainty in such data is preventing the successful application of cermets in rockets and missiles.

As the temperatures of rocket parts reach higher extremes, the search for materials that resist chemical erosion and maintain their mechanical properties at these temperatures has been intensified. Refractory brittle materials, such as cermets, appear to meet this need, but the lack of reproducible data on their mechanical properties introduces uncertainty in the design and processing of parts. Consequently, failures in performance are more apt to occur, making the missile unsafe or ineffective.

Obviously, all the properties of a given specimen, if properly determined, should be compatible. For example, Young's modulus of elasticity should be the same and should stand in the established relation to other elastic properties whether it is obtained from tensile, compressive, or transverse tests. To make possible such agreement, the present work was undertaken by M. J. Kerper, L. E. Mong, M. B. Stiefel,[2] and S. F. Holley of the refractories laboratory.

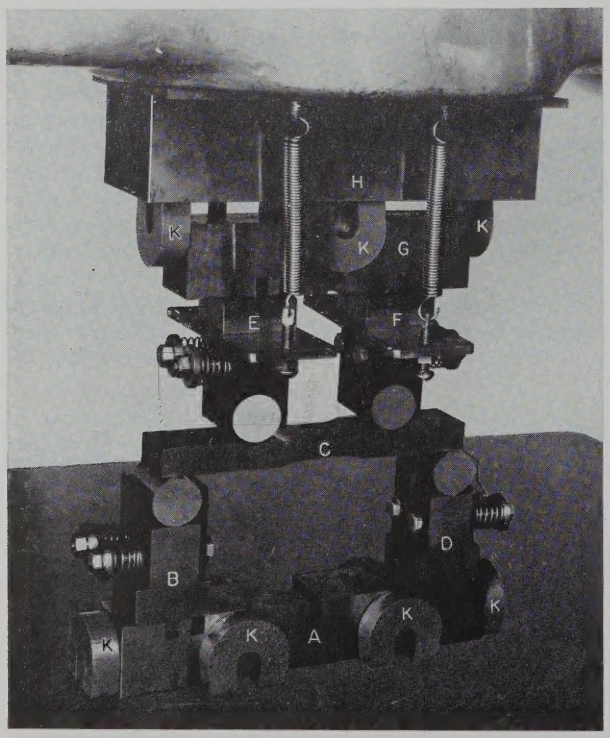
The disagreement among experimental results for ceramics is attributed to many causes. Extremely important is the brittleness of cermets, which makes it necessary to refine the procedures and improve the apparatus used to study ordinary ductile materials. Also, in ceramic materials, correct distribution of stress is sometimes difficult to obtain, and extraneous stresses are easily introduced.

These factors, which limit accuracy, were given special consideration so that errors will not appear in the final strain-gage determinations. For example, in the tensile tests, several specimen designs, differing mainly in the provision made for applying load, were compared. One of these, the pin-end design (with a hole at each end to allow mounting)—previously considered adequate for tensile tests—was found unsatis-



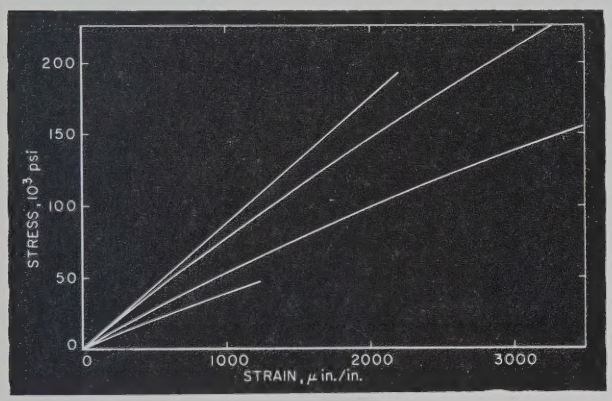
Specimen types used in tensile testing of brittle cermets to evaluate test methods. Typical fractures: Pin-end (A), T-end (B), shoulder-end (C), and gripped-end (D) specimens.

Loading apparatus used in the transverse testing of enlarged-end brittle-cermet specimens. Block A spaces supports B and D for specimen C. Load, applied by the crosshead H, is transmitted to beam G and then to the specimen. D, E, and F have rocker bases to eliminate extraneous stresses in C. The magnets K position parts.



factory because of excessive stress concentrations. Although other designs were suitable, the final correlation of the results from all types of tests shows that best results are obtained with specimens grooved at the ends, the gripped-end design.

A particular type of mounting arrangement was adopted for compressive tests because of the difficulty in securing satisfactory alinement. The usual ways employed to secure alinement of the specimen—spherical bearings or a cast-in-place layer of plaster of Paris—gave a nonuniform distribution of stress; in some cases, one side of the specimen was actually in tension. Successful alinement was secured with specimen having parallel ends set between flat bearing blocks on anvils to which the load is applied. To eliminate another source of error, very hard bearings were used, preventing specimen penetration. These were reground after each test to remove roughness.



Stress-strain curves obtained in tensile tests of four specimens of the gripped-end design. Stress-strain curves for transverse, compressive, and torsion tests are similar to those for tensile tests.

To assure that the stresses being measured in torsional tests are due to twisting only, a special machine is employed. In other tests of this type, bending of the specimen is often present to some degree, making results inaccurate. Such extraneous causes of stress are eliminated by a frame that is symmetrical about the specimen axis.

An apparatus to prevent undesirable stresses was designed for the transverse test also. Here rocker bases eliminate stresses due to causes other than simple bending.

The correlation of results from the modified tensile, compressive, torsional, and transverse tests indicate that elastic properties can be obtained satisfactorily from each. Poisson's ratio is the exception, being too low for stubby transverse specimens.

A comparison of the four tests shows that all are approximately equal in complexity and cost. The tensile test has the advantage of yielding tensile strength directly without corrections for plasticity or creep. The compressive test has the same advantage in shear-strength determinations. Thus, these two tests are preferable for the determination of significant mechanical properties.

On the other hand, the transverse and torsional tests give the transverse and torsional strengths, and are most useful in design problems which include bending and twisting applications. The tensile strength may also be calculated from the transverse and torsional strengths by making corrections based on surface-strain measurements.

In addition to the tests mentioned, impact tests were conducted and were found to give some indication of the performance of materials subjected to sudden loading. As the energy causing fracture is only a small fraction of that which can be stored without fracture at low-loading rates, stress concentrations are thought to be excessive. Although several correlations of factors derived from mechanical properties were attempted, the impact value could not be predicted with any accuracy from other tests.

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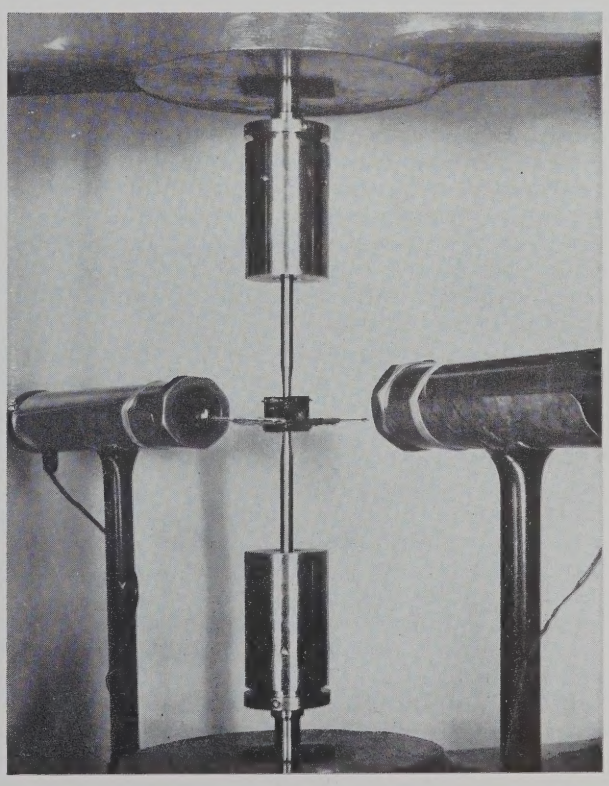
[1] Evaluation of tensile, compressive, torsional, transverse and impact tests and correlation of results for brittle cermets, *by M. J. Kerper, L. E. Mong, M. B. Stiefel, and S. F. Holley, J. Research NBS* **61**, *149 (1958) RP2895.*

[2] *Presently with Arthur, Dry, and Doyle. Rockefeller Center, New York, N. Y.*

Tensile testing of a gripped-end specimen in evaluation of test methods for brittle cermets. Strain is measured by the optical gage, mounted on the reduced portion of the specimen, and read through collimators at right and left.



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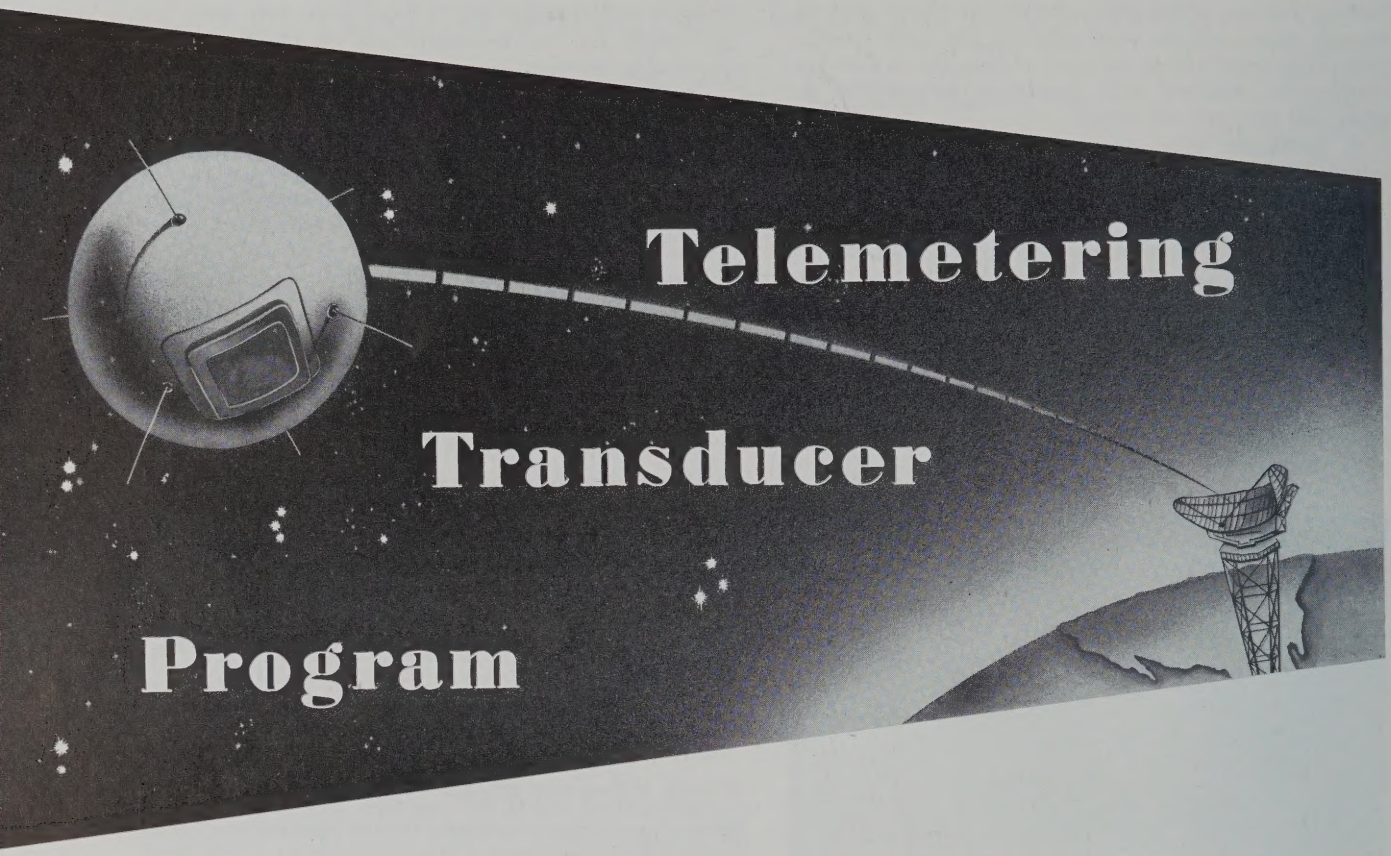


A PROGRAM on telemetering transducers is being conducted by the Bureau for the Navy Bureau of Aeronautics, the Air Force, and the Army Ordnance Corps. Carried on in conjunction with complementary programs in measurements, this effort involves the development of test methods pertinent to the selection, application, and improvement of transducers. Data on the inherent characteristics of the principal types of transducers as well as a better understanding of the requirements for various applications have thus far resulted from the study.

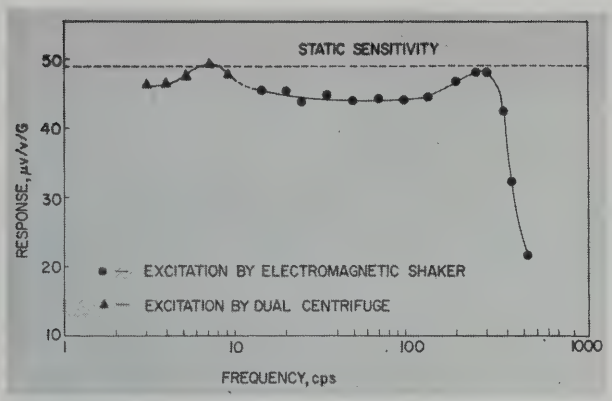
Transducers—electromechanical devices for converting pressure, displacement, acceleration, force, and similar mechanical quantities to electrical signals—are widely applicable, but are in greatest demand for missile and aircraft-flight testing. In such use, the electrical output of a transducer is transmitted by a wire line or radio-telemetering link to a recording device. However, a serious drawback to the use of these telemetering systems is the limiting performance of the transducer itself. To provide adequate data on instrument performance, and establish a satisfactory basis for transducer testing and selection, the Bureau undertook the present work.

In any evaluation of missile and aircraft-flight-test instruments, the environmental conditions encountered in use are of basic importance. The transducer program, directed by E. C. Lloyd, Chief of the Mechanical Instruments Section, provides data on the performance of transducers over significant ranges of ambient conditions. The conditions imposed are temperature, temperature shock, pressure, vibration, and acceleration; extremes of humidity, pressure shock, or acoustic vibration are included in cases where their effects may be important.

In addition to the general performance studies, attention has been focused on special problems arising in certain transducer applications. Such problems appear, for example, in pressure and acceleration measurements.

In missile and aircraft-flight testing, more measurements are made of pressure than of any other quantity. One serious limitation on the use of transducers in this area has been lack of information on their dynamic performance. To obtain such data, a shock tube—designed and built for this purpose under the supervision of R. O. Smith and P. S. Lederer—has been employed. This apparatus provides a pressure step-function of known value, which can be applied to the transducer pressure gage under study; the response of the gage is then detected by an oscilloscope and photographically recorded for further analysis.

In further studies of dynamic properties, considerable effort has been expended on the testing of transducers employed as accelerometers. Calibration of



Frequency response of a strain-gage accelerometer. The Bureau employs a dual centrifuge to calibrate accelerometers at low frequencies and high amplitudes not available with the usual electromagnetic shaker method.

such instruments presents no difficulties at frequencies from 10 to several hundred cps. However, the frequency of interest often lies below this—a region where satisfactory test equipment has not been available. Two methods have therefore been devised for calibrations at lower frequencies. One procedure entails rotation of the accelerometer in the earth's gravitational field. The other makes use of a specially designed dual centrifuge. Either provides a known frequency input to the accelerometer under test; a response curve then represents, in the most easily read fashion, the dynamic characteristics of the device.

Such studies of selected transducers have yielded a considerable body of knowledge concerning the inherent characteristics of various types of transducers as well as an understanding of requirements in the application of transducers. At present, the number of transducers being offered by manufacturers requires a substantial effort merely to keep abreast of development. Future activity under the transducer program will include continuation of test-method development, measurement of transducer performance, and a review of the characteristics of transducers of foreign manufacture.

The dynamic testing of pressure transducers yields results in terms of response to step-function inputs. This manner of determining dynamic characteristics was selected because step-function inputs of known pressure and over a wide range of amplitude are readily produced. On the other hand, obtaining accurately known sinuosoidal inputs in the form of pressure is difficult, except for very small amplitudes. However, the ease with which step-input responses can be interpreted led to work by the Bureau's data-processing laboratories to explore analog-computer techniques for deriving frequency-response characteristics directly from step-function response records.

Shock Tube

The shock tube was used because it is the most effective method of generating step-function pressure changes of known amplitude. Constructed of flanged steel sections, it has a working cross-sectional area 3 in. square, a compression chamber variable in length from 3.4 to 12 ft, and an expansion chamber 8 ft in length. The expansion-chamber sections are carefully

To fire shock tube, the diaphragm between compression and expansion chambers (shown being inserted at left) is burst. A camera, focused on an oscilloscope screen, photographs pressure-gage response.

Mounting an accelerometer on a dual centrifuge for dynamic calibration.

finished and chrome-plated on the interior surfaces to minimize "noise" due to shocks reflected from surface discontinuities. The compression-chamber sections move on rollers to permit insertion of diaphragms.

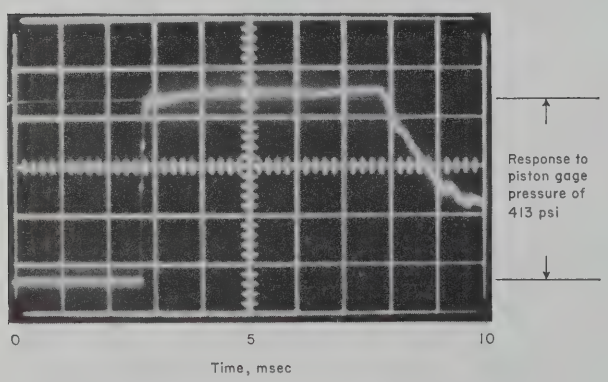
The tube is equipped with three pairs of optical-glass windows, each pair provided with a schlieren system and photomultiplier tube arranged to give a pulse at the time of passage of the shock wave. Auxiliary equipment is used to measure accurately the initial expansion-chamber pressure, and to determine the transit time of the shock wave between schlieren stations.

The tube provides pressure steps lasting 4 to 5 msec. Compression-chamber pressures up to 1,000 psi and expansion-chamber pressures up to 350 psi can be selected. By using air on both sides of the diaphragm, shock-step heights may be generated up to about 600 psi. The use of helium in place of air in the compression chamber at initial pressures up to 1,000 psi produces shock-step heights up to 1,000 psi. The amplitude of the pressure step is determined from the measured value of the initial expansion-chamber pressure and temperature and the shock-wave velocity. The rise time of the step is less than 10^{-8} sec.

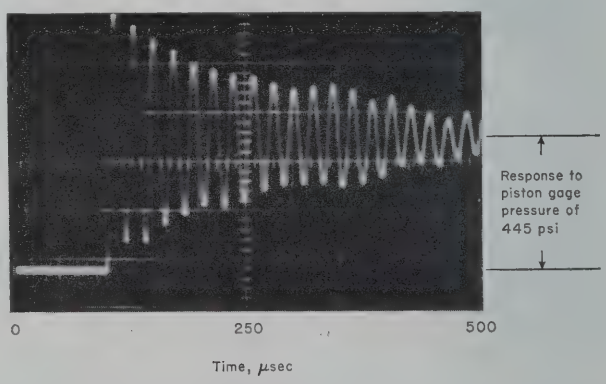
Dual Centrifuge

The dual centrifuge consists of a rotating table carrying a second smaller rotating table to which the accelerometer is fastened. Both tables rotate in horizontal planes with such a speed that the test accelerometer is carried around a circular path without rotation. Such an arrangement provides a sinusoidal input for the accelerometer, and allows electrical connections to be made directly to the accelerometer. The dual centrifuge has a useful range of frequency from ½ to 10 cps, and a displacement that is variable up to ±11¼ in. The corresponding acceleration amplitude is ±25 g at 5 cps and about ± 1 g at 1 cps.

The vertical-rotation procedure allows accelerometer calibration from 0 to 30 cps, but limits the acceleration range to ±1 g and makes necessary the use of slip rings for electrical connections.



Record obtained from a quartz-crystal-type pressure transducer. The amplitude of the pressure step, supplied by a shock tube, was 413 psi, and the response of the gage to a static pressure of 413 psi is shown at right for comparison.



Oscilloscope record obtained by use of the shock tube from a flush-diaphragm-type pressure pickup. Amplitude of the pressure step applied to the gage was 445 psi and response of the gage to a static pressure of this value is shown at the right for comparison.

Glasses With High Refractive Indices and Dispersions

BECAUSE of its extensive research on the properties of glasses and glass systems, the Bureau was requested to undertake the development of special lightweight optical glasses with high refractive indices and high dispersions for the Navy Bureau of Ordnance.[1] Because of their optical properties, these glasses may be used as elements of compound lenses, particularly in applications such as aerial photography and periscopes where a wide field of view with minimum distortion is essential. In such systems, elements of these glasses would be advantageous for eliminating astigmatism, coma, and chromatic and spherical aberrations. The low densities of the glasses make them especially suitable for use in aircraft where weight is of prime importance.

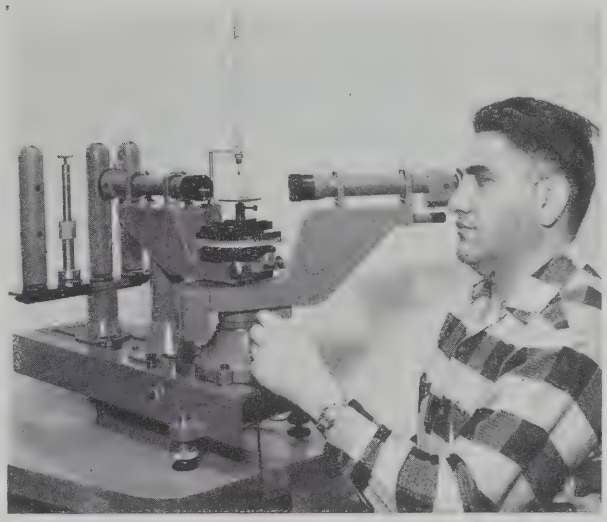
Usually, optical glasses with high refractive indices and high dispersions contain lead oxide as the principal constituent. However, since increasing the lead content of such glasses increases the refractive index, it also places a limiting value on the corresponding dispersion. As glasses are needed in which both the refractive indices and dispersions can be increased beyond the present limits, work aimed in this direction was undertaken in connection with basic research on the properties of glass-forming systems.

As a starting point for the study, use was made of the fact that glasses containing appreciable quantities of titanium oxide have high refractive indices and dispersions. The sodium titanium silicate system was investigated in detail by E. H. Hamilton and G. W. Cleek of the glass laboratories, and found to produce glasses with the desired optical characteristics.

Glasses were made which contained from 10- to 45-mole percent Na_2O, 0- to 40-percent TiO_2, and 30- to 85-percent SiO_2. For such compositions, refractive indices range from 1.5184 to 1.8005, and v-values—inverse functions of dispersion—from 51.5 to 23.2. The refractive indices are comparable to those exhibited by lead-containing or flint glasses; and, as desired, the dispersions are higher than for flint glasses at corresponding high indices.

For example, the titanium glass with a refractive index of 1.7184 had a v-value of 27.6, while extra-dense flint glass with an index of 1.7200 has a v-value of 29.3. The lower v-value of the glass under investigation is indicative of its greater dispersion. This dispersion would be even higher if the refractive index of the investigated composition were increased to equal that of the flint glass. The increase in refractive index is effected by raising the ratio of TiO_2 to SiO_2, or TiO_2 to Na_2O.

Composition has an effect on physical as well as optical properties. All glasses containing 5-mole percent of TiO_2 and some containing 10 and 15 percent of this substance were practically colorless, while all other compositions were either amber or yellow. The darker glasses are, of course, not suitable for use in optical instruments, except possibly as filters. However, increasing the ratio of Na_2O to SiO_2 reduces the amount of color, making the sodium titanium silicate glasses compare favorably with lead-containing glasses in this respect also.



Measuring the index of refraction of one of the highly refractive, highly dispersive glasses developed at the Bureau.

The density of the titanium glasses is significantly lower than that of lead glasses with comparable properties. Specific gravities of the recently developed glasses range from 2.12 to 3.00, compared with 3.0 to 5.0 for the lead-oxide glasses.

Before these glasses are actually employed as lenses, filters, or windows, certain modifications in composition will be necessary. For example, constituents must be introduced to compensate for devitrification in some glasses or for hygroscopicity in others. With these relatively simple additions, the sodium titanium silicate system can be employed to manufacture glasses with refractive indices and dispersions previously unattainable.

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[1] Properties of sodium titanium silicate glasses, *by E. H. Hamilton and G. W. Cleek, J. Research NBS* **61,** *89 (1958) RP2890.*

TELEVISION COLOR STANDARDS

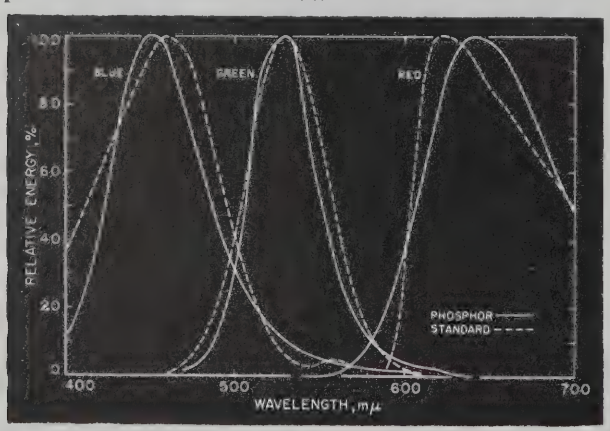
BRIGHTNESS STANDARDS for color-television tubes have been made available by the Bureau in cooperation with the Joint Electron Tube Engineering Council of the Electronic Industries Association (formerly RETMA). Each set consists of three standards—red, green, and blue—closely matching in spectral energy the three phosphors which in the tube act together to produce various colors in the image. The standards are used to calibrate instruments for measuring the color and brightness of the phosphors. They thus provide a simple, accurate means for achieving uniform color reproduction in television tubes.

The design and calibration of the standards were performed by M. A. Belknap, V. I. Burns, D. B. Judd, and R. P. Teele of the Bureau staff. Data on the spectral-energy distributions emitted by the phosphors used in color television tubes were supplied by a subcommittee of the Council. From these data one set of standards was designed and built. These are retained at the Bureau as master standards and are used to calibrate the standards supplied to industry.

In order to match the spectral energies of the phosphors as closely as possible, a light blue-green glass and a dark-blue glass are used as filters for the blue standard; a sextant-green glass for the green standard; and a traffic-red glass combined with a blue-green glass for the red standard. Each of these filters or filter combinations is fitted with an opal glass that diffuses light from a calibrated 500-w projection lamp furnished as one of the components of each set of standards. A comparison of the lamp with national color-temperature standards maintained by the Bureau showed that light with the desired color temperature of 2,854° K is transmitted through the opal glass when the lamp is operated at 2,916° K.

Reference standards for achieving color and brightness uniformity in color television. The three diffusing glass-filter combinations and a 500-w projection lamp are carefully packaged for shipment.

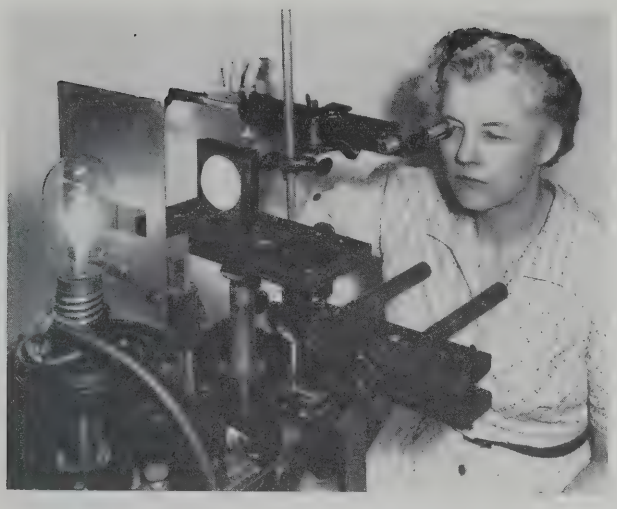
Relative spectral energy distributions of the three phosphors and the three standards.



The luminous transmittances of the filters in the master standards—that is, the fraction of incident light they transmit—were measured with a physical photometer and a recording spectrophotometer. The filters were then fitted to the opal glass diffusers and the luminance or photometric brightness of each was computed from the luminous transmittance of the colored filters, and the measured luminance of the opal glass. When additional filters and opal glass diffusers were cut and mounted, their luminances were established by photoelectrical comparison with the master set.

The standards can be used for calibrating either photoelectric or visual photometers. After calibration, the photometer may be used to measure the luminance of the color-television tube.

Prior to calibration, the lamp should be positioned on a photometer bar and allowed to warm up for about 5 minutes. During this time, the filters should be shielded from the lamp's radiant energy since luminous transmittances, especially of the red filter, undergo a change when the filters are heated, causing a decrease in luminance of the standard.



Calibrating a portable photometer used to measure brightness of color-television tubes. The 500-w projection lamp has been positioned on a photometer bar at the proper distance from the masked opal glass filter of the standard to produce a luminance of 50 fL.

A test was made to find out just how much the luminance of the standards diminished during a 20-min exposure to the lamp. The table gives the results obtained when the voltage and position of the lamp were adjusted to produce a luminance of the standard of 50 or 20 fL. When the lamp was positioned at a distance from the opal glass required to produce 2 fL, the heating effect was found to be negligible.

Instructions for calibrating photometers, provided with each set of standards, give the distances required between the opal glass of the standards and the lamp, and the lamp voltage necessary for the standards to have luminances of 50, 20, and 2 fL, respectively. These particular values of luminance were chosen by the Council as the most useful for industrial purposes. However, the conditions required to produce other luminances less than 50 fL can be easily computed.

Sets of the standards are available from the Photometry and Colorimetry Section, National Bureau of Standards, Washington 25, D. C., at $250.00 a set. To facilitate the use of these standards, a detailed report of their design and calibration is included with each set.

TABLE 1. *Decrease in luminances of standards after exposure to projection lamp*

Standard	Foot-lamberts	Percentage-decrease per minute
Blue	50	0.25 for first 11 min. .16 for next 9 min.
	20	.19 for first 8 min. .15 for next 12 min.
Green	50	.39 for first 8 min. .22 for next 12 min.
	20	.20 for first 8 min. .06 for next 12 min.
Red	50	.83 for first 7 min. .44 for next 6 min. .23 for next 7 min.
	20	.55 for first 4 min. .31 for next 6 min. .15 for next 10 min.

Determination of Acetylene in Low Concentrations

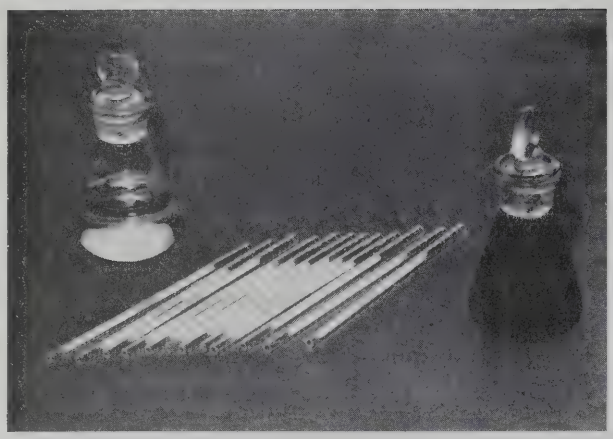
method useful in air pollution control

A RAPID, accurate, colorimetric method for detecting and measuring minute quantities of acetylene in air has been developed by the Bureau in cooperation with the U. S. Public Health Service. Since acetylene in the air is associated with the contaminants from automobile exhaust gases, the method is expected to be useful in the study and monitoring of urban air pollution. The process,[1] developed by E. E. Hughes and R. Gorden, Jr., gas analysis laboratory, permits detection of acetylene concentrations as low as 10 parts per billion. The equipment required is simple and portable, so that the method is well suited for field studies.

The first step in the procedure is to concentrate a measurable amount of the dispersed acetylene by adsorption on silica gel. The quantity of acetylene present is indicated by the depth of color produced when the silica-gel surface is treated with a solution of ammoniacal cuprous chloride. Then the exact concentration is determined by visually comparing this color with those produced by gas samples containing known concentrations of acetylene.

Urban Air Contaminants

A normal urban atmosphere contains a wide variety of gaseous inorganic and organic contaminants. These come mainly from industrial processes and vehicles—the internal combustion engine is a major source of the organic contaminants. Although the quantities of water vapor and oxides of nitrogen and carbon in exhaust gases have been determined, the organic fraction of "traffic gas" has never been completely analyzed. This remainder contains numerous hydrocarbons whose individual concentrations are too low for quantitative detection by previously available methods. The development of rapid procedures for the qualitative or quantitative determination of such substances has become of great importance for the early detection of changes in pollution patterns of certain areas. In particular, the analysis of certain substances—such as acetylene—which have been found in relatively constant concentrations in traffic gas, serves as a simple and practical means of determining the degree of contamination.



After acetylene samples have been collected on silica gel and treated with reagent solution (flasks, left and right), they are visually compared with samples (center) prepared from known concentrations of acetylene. Samples range in color from pink to brown, depending on acetylene concentrations.

Besides its use in the study of air pollution, this method can be applied to other industrial or laboratory processes requiring the determination and fractionation of air constituents. For example, accumulation of solid acetylene in liquid-air generators has caused numerous costly and dangerous accidents. Although the permissible level of acetylene in the intake air is quite low, it can easily be detected by this procedure.

Procedure

To begin the determination, a definite volume of air contaminated with the acetylene is drawn through a long, small-diameter glass tube containing highly purified silica gel. As the air sample passes through the tube, which has been cooled to the temperature of dry ice, acetylene is adsorbed on the gel's surface. A column of fine glass beads bounds the gel layer at each end of the tube, making possible easier detection of slight color changes. Because the acetylene collects mainly on the initial portion of the gel, the tube is allowed to warm to room temperature for a short time to obtain a homogeneous distribution. An ammoniacal solution of cuprous chloride—obtained by mixing a solution of ammoniacal cupric chloride with hydroxylamine hydrochloride—is then introduced into the tube and the copper acetylide formed precipitates directly on the gel surface. The resulting color ranges from pink to brown, depending on the quantity of acetylene.

When the concentration of acetylene in the atmosphere is 10 ppm or above, detectable quantities can be adsorbed at room temperature. At concentrations below 10 ppm, the gel must be cooled to $-78°$ C, the temperature of dry ice. Because of increased adsorption at this temperature, it has been possible to detect concentrations as low as 10 parts per billion.

The concentration of acetylene in the unknown air sample is determined by comparing the color of the unknown with that of a tube exposed to a sample of known concentration. Mixtures of acetylene and air for the comparison samples are made by successive dilution of a single gas mixture containing 1 percent acetylene.

Acetylene collected in the field can either be treated with the reagent immediately, or stored at a reduced temperature in liquid nitrogen and then returned to the laboratory for analysis. The final analysis does not have to be made immediately, because the collected acetylene which has been treated with the cuprous-chloride solution can be stored for at least 48 hr without apparent loss of color.

In general, experiments indicate that this method can identify total adsorbed quantities of acetylene as low as 0.01 μg contained in 12 ml of air at a volume concentration of 1 ppm. To detect an equal amount of acetylene at a concentration of 0.1 ppm, it was only necessary to sample a volume of air 10 times greater. Differences of 0.012 μg between specimens of acetylene were easily observed in the range of 0.00 to 0.237 μg.

[1] *For further technical information, see* A simple method for the determination of acetylene in air in concentrations from one part per million to ten parts per million, *by E. E. Hughes and R. Gorden, Jr., Anal. Chem. (in press).*

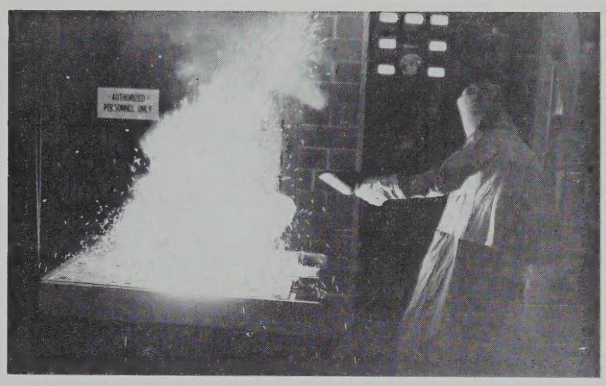
Compact, portable equipment used to rapidly collect and analyze small amounts of acetylene dispersed in air.

IRON-BASE SPECTROSCOPIC STANDARD SAMPLES

A SET of eight new standard samples of ingot-iron and low-alloy steel is now available from the Bureau.[1] Analyzed and certified for 17 elements, these standard samples [2] are designed for control of composition in the iron and steel industry and for analysis of the finished products by both the producer and the consumer.

The new samples are the first spectroscopic standards which the Bureau has certified for carbon and phosphorus—two elements that can now be determined spectroscopically—increasing the speed and efficiency of the complete analysis. The samples are also the first to be issued in a new-size disk in addition to the usual rod form, which is 7/32 in. in diameter and 4 in. long. The new disk, 1¼ in. in diameter and 3/4 in. thick, is suitable for X-ray fluorescence analysis as well as optical-emission spectroscopy.

In combination with other ingot-iron and low-alloy steel standard samples already available from the Bureau, the present set of standards provides certified concentration values for determining most of the elements encountered in the analysis of iron and low-alloy steel samples. Certification has been made for the following elements: carbon, manganese, phosphorus, silicon, copper, nickel, chromium, vanadium, molybdenum, tin, titanium, boron, arsenic, tungsten, zirconium, niobium, and tantalum. Several additional elements may be certified later.



Powdered carbon is added to the induction furnace to remove excess gases from the molten base material prior to the addition of alloys.

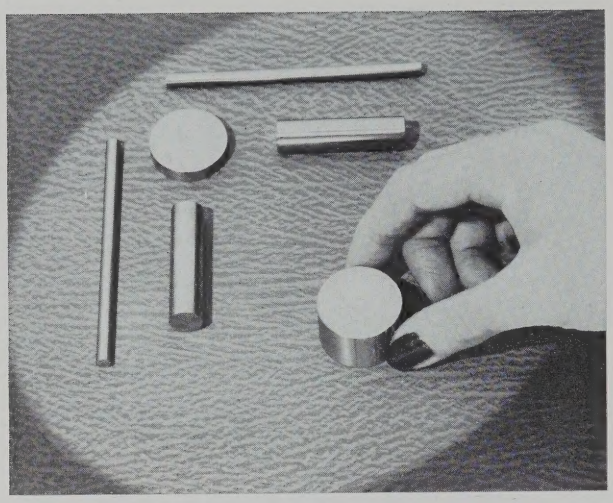
Before preparing the samples, melting, casting, fabricating, and testing procedures were investigated to insure proper graduation of concentration ranges and high homogeneity in the final product. For each standard the material was prepared by melting in a 1-ton induction furnace, removing excess gases, adding alloying elements, and introducing rare earths as a possible aid to homogeneity. Then the material was processed by forging the ingots into slabs, discarding portions of questionable homogeneity, hot-rolling selected material to oversized rods, annealing, and centerless grinding to size.

The standards can be applied directly to the analysis of steel rods and disks prepared in a similar manner and with similar dimensions. However, when the standards are applied to the analysis of steel samples prepared by different methods, adjustments may be required to correct deviations caused by physical differences.

TABLE 1. *Spectrographic ingot iron and low-alloy steel standards*

NBS No.[a]	461 1161	462 1162	463 1163	464 1164	465 1165	466 1166	467 1167	468 1168
Designation	Low-alloy steel A	Low-alloy steel B	Low-alloy steel C	Low-alloy steel D	Ingot iron E	Ingot iron F	Low-alloy steel G	Low-alloy steel H
Element	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Carbon	0.15	0.40	0.19	0.54	0.037	0.065	0.11	0.26
Manganese	.36	.94	1.15	1.32	.032	.11$_3$	.27$_8$	.47
Phosphorus	.053	.045	0.031	0.017	.008	.012	.033	.023
Silicon	.047	.28	.41	.48	.029	.025	.26	.075
Copper	.34	.20	.47	.094	.019	.033	.067	.26
Nickel	1.73	.70	.39	.13$_5$	.026	.051	.088	1.03
Chromium	0.13	.74	.26	.078	.004	.011	.036	0.54
Vanadium	.024	.058	.10	.29$_5$	.002	.007	.041	.17
Molybdenum	.30	.080	.12	.029	.005	.011	.021	--------
Tin	.022	.066	.013	.043	.001	.005	.10	.009
Titanium	-----[b]	.037	.010	.004	.20	.057	.26	.011
Boron	.000$_2$	.000$_8$	.0012	--------	.000$_1$	--------	--------	--------
Arsenic	--------	.046	.10	.018	.010	.014	--------	.008
Tungsten	.012	.053	.19$_5$	.022	--------	--------	.20	.077
Zirconium	--------	.063	--------	.010	--------	--------	.094	--------
Niobium	.011	.096	.19$_8$	.037	--------	--------	.29	--------
Tantalum	.002	.036	.15	--------	.001	--------	.23	--------

[a] 400 series, rods 7/32 in. in diameter and 4 in. long; 1100 series, disks 1¼ in. in diameter and ¾ in. thick.
[b] Dashes indicate elements not certified for spectrographic analysis.



Iron-base spectroscopic standard samples recently made available by the Bureau. The flat disk form at right foreground (1¼ in. in diameter and 3/4 in. thick) is suitable both for optical and X-ray spectroscopic analysis and will be the established form for other new standards.

The rod and disk samples for this set of standards may be obtained from the National Bureau of Standards for fees of $4.00 and $6.00, each, respectively.

[1] *The National Bureau of Standards distributes 600 different, certified standard samples of chemicals, ores, ceramics, and metals. Additional information is contained in* Standard Samples, *NBS Circular 552 (second edition), which may be obtained by writing to the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.*

[2] *For further information, see* Recent developments in NBS spectrographic standard samples of iron-base alloys, *by R. E. Michaelis, Applied Spectroscopy (in press).*

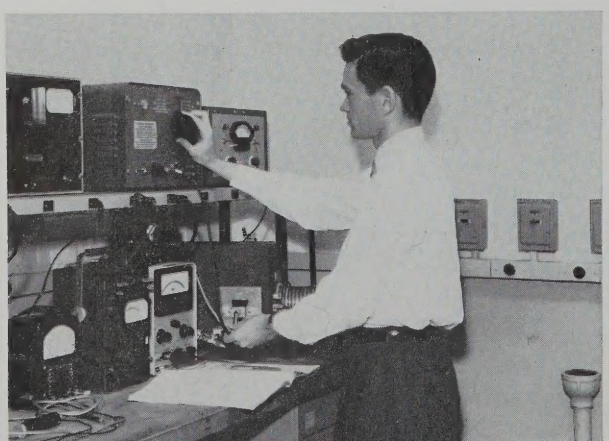
Electroluminescence from Barium Titanate

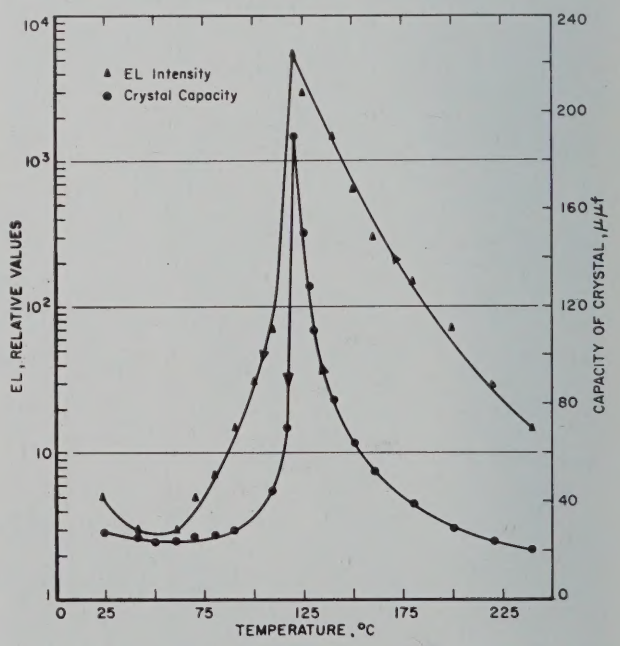
ELECTROLUMINESCENCE from the surface layer of barium titanate has been observed at the Bureau and is now being used as a tool to study the material itself.[1] The light emission, which is extremely low in both intensity and efficiency, occurs when a radiofrequency voltage is applied to the $BaTiO_3$ sample. Data obtained from the emitted light have led to an understanding of the mechanism by which the light is generated. This, in turn, provides knowledge about the unusual surface layer of barium titanate.

Barium titanate is used extensively in electronic devices because of its desirable dielectric and piezoelectric properties. Important applications of this material include high-capacity miniature capacitors, and transducers used in accelerometers, sound detectors, and ultrasonic transmitters. In addition, $BaTiO_3$ memory elements for use in computers are now in the developmental stage in many laboratories. However, these memory units, at present, tend to fatigue or "forget". The fact that different types of electrodes have a direct influence upon this characteristic—a dependence which implies a surface effect—has emphasized the need for surface-layer studies. The Bureau's work on $BaTiO_3$ surface-layer electroluminescence is being carried on in the electron devices laboratory by G. G. Harman and R. L. Raybold.

To produce light emission from the surface of $BaTiO_3$, the most satisfactory arrangement is a concentric con-

Measuring the capacity of a barium titanate crystal (mounted in holder above scientist's left hand) in preparation for studying the electroluminescent spectral emission.





Emission intensity and crystal capacitance of barium titanate plotted against temperature. Electroluminescence from the surface of the crystal increases in intensity by a factor of about 1,000 at the Curie point.

figuration of the electrodes. This concentrates the rf field around the small center electrode and produces maximum light output. Although various metals serve successfully as electrodes, best results are obtained from either indium-gallium rubbed directly on the crystal surface or evaporated silver, soldered with indium.

The studies conducted with this arrangement show that at the Curie point [2] of $BaTiO_3$ the light-emission intensity jumps to about 1,000 times its value at lower temperatures. This effect is attributed to the fact that at the Curie point the surface dielectric constant remains stable while the bulk dielectric constant increases by a factor of 8 or 10. Thus, more voltage is applied across the surface layer, causing an increase in intensity. A similar phenomenon occurs at frequencies corresponding to piezoelectric resonances. The resonances also produce increases in light emission because the bulk properties change while the surface properties remain stable.

The emitted light is generally yellow, but its color depends somewhat on the type of electrode employed. Spectral-emission data show that light is emitted from 7000 A to 3000 A—indicating photon energies as high as 4 ev. Such energies are considerably above the 3-ev optical-absorption edge of the $BaTiO_3$ crystal. The theory introduced to account for light emitted with energies higher than expected is the "field emission" or the "avalanche breakdown" mechanism.[3] The electrons necessary for electroluminescence in $BaTiO_3$ come from the metallic electrode to the adjacent surface layer by field emission.

In further studies, crystals were cut into cylindrical pieces for electroluminescence measurements. This geometry allowed the ratio of permittivity to thickness of the $BaTiO_3$-surface layer to be determined. Since the surface-layer thickness was already known to be about 10^{-5} cm, the permittivity (ϵ) itself could be assigned a value. This value, $\epsilon=5$, is equivalent to the optical dielectric constant and is considerably smaller than the average bulk dielectric constant, which is approximately 1,000.

---

[1] Light emission from the surface layer of $BaTiO_3$, $SrTiO_3$, and associated materials, *by G. G. Harman, Phys. Rev.* **111**, *27, July 1, 1958. See also* Electroluminescence from $BaTiO_3$ single crystals, *by G. G. Harman, Bul. Am. Phys. Soc., Ser II,* **1**, *112 (March 15, 1956).*

[2] *The Curie point is the temperature above which ferroelectric materials lose their spontaneous polarization.*

[3] *For further technical details, see* Photon emission from avalanche breakdown in silicon, *by A. G. Chynoweth and K. G. McKay, Phys. Rev.* **102**, *369 (April 15, 1956).*

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A. V. ASTIN, *Director*

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